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ABSTRACT

Applications at synchrotron facilities such as macromolecular crystallography and high energy X-ray diffraction require high resolution imaging detectors with high dynamic range and large surface area. Current systems can be split into two main categories: hybrid pixel detectors and scintillator-coupled Charge-Coupled Devices (CCDs). Whilst both have limitations, CCD-based systems (coupled to fibre-optics to increase imaging area) are often used in these applications due to their small pixels and the high resolution. Electron-Multiplication CCDs (EM-CCDs) are able to suppress the readout noise associated with increased readout speed offering a low noise, high speed detector solution. A previous pilot study using a small-area (8 mm × 8 mm) scintillator-coupled EM-CCD found that through high frame-rates, low noise and novel uses of photon-counting, resolution could be improved from over 80 μm to 25 μm at 2 fps. To further improve this detector system, high speed readout electronics can be used alongside a fibre-optic taper and EM-CCD to create a "best of both worlds" solution consisting of the high resolution of a CCD, along with the low noise, high speed (high dynamic range) and large effective area of pixel detectors. This paper details the developments in the study and discusses the latest results and their implication on the system design.

Keywords: CCD, EM-CCD, high speed, low noise, crystallography, tomography, scintillator

1. INTRODUCTION

Many applications in synchrotron research, from imaging uses in macromolecular crystallography and high-speed tomography to dispersive spectrometry, require imaging of X-rays. A wide variety of detectors are available for such applications, but those with imaging capabilities can be split into two main categories: scintillator-coupled Charge-Coupled Devices (CCDs) and hybrid pixel detectors.

Hybrid pixel detectors, such as the Dectris Pilatus [1] or Medipix series of detectors [2], often have larger pixels than their CCD-based counterparts but are able to operate at high speed with low noise. The hybrid detector is normally formed from a CMOS readout chip coupled to a detector layer, either made from silicon or an alternative semiconductor material. Operating modes are often able to offer effectively zero noise by using thresholding on the signal, although this severely limits any spectral capabilities which are only available in this case through methods such as time-over-threshold. Modern advances have allowed the size of the pixels to decrease, bringing new applications to detectors of this type, however, the nominal operation mode of hybrid detectors does not allow the same use of centroiding (centre of mass calculations to determine the sub-pixel location of an event) as that available using a CCD-based system.

Scintillator-coupled CCDs make use of fibre-optic tapers or lens systems to couple a scintillating layer to the CCD [3,4]. The taper or lens system allows magnification to be added to the camera, effectively increasing the active imaging area, and helps to prevent direct detection of higher energy X-rays in the CCD. The small pixel sizes available with CCD-coupled systems allow much higher resolutions to be achieved. The scintillating layer can be adapted to the application and X-ray energy range in question, with a thicker scintillation layer giving higher detection efficiency but decreased spatial resolution, and a thinner scintillation layer giving lower detection efficiency but higher spatial resolution. There are, however, limitations in CCD-based systems caused by the increase in the readout noise as the readout speed of the system is increased. Centroiding with increased noise levels is not possible and therefore the resolution and/or the readout speed of the detector are limited.

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Over recent years we have demonstrated a proof-of-principle camera using a small area Electron Multiplying (EM) CCD, operated at 2 fps, coupled to a scintillator through a 1:1 fibre-optic plate [5-9]. The EM-CCD is similar in structure to the CCD but includes an additional register between the standard readout register and the output node: the multiplication gain register [10]. Through the introduction of impact ionisation in this multiplication gain register, the signal can be increased before it is read out of the device and therefore before the readout noise is added. This increase in signal without an increase in the readout noise allows effective readout noise levels to be achieved at the sub-electron level, even at high readout speeds. With this proof-of-principle system it was shown that is was possible to improve the integrated image spatial resolution from 80 µm down to 25 µm using centroiding techniques, whilst also introducing the possibility to provide basic spectral information through novel analysis techniques. The small area of 8 mm × 8 mm and relatively slow frame-rate, despite acting as a suitable proof-of-principle, dramatically reduce the number of potential applications for the detector and we therefore aim to produce a large-area, high-speed proof-of-principle detector system

With the use of a high-speed camera drive system [11], we are developing a scintillator-coupled EM-CCD camera system that operates at 25 MHz, offering 120 fps (full-frame) with a 512 × 512 pixels imaging area as with the detector used in the previous study, or at up to 30 fps (full-frame) with a 1024 × 1024 pixel detector. The system can still operate with sub-electron noise, even at high readout speeds, and the noise introduced by the gain process is no higher than that achieved with a gain level of approximately ten to twenty times.

Following a brief introduction to the EM-CCD and a historical overview of the proof-of-principle camera system that lead to the latest developments, we present the first details of the camera and outline the development of the new proof-of-concept high-speed, larger area, modular system from here forwards.

2. ELECTRON-MULTIPLYING CCD (EM-CCD)

The Electron-Multiplying CCD is similar in design to the standard CCD except for the addition of a multiplication gain register, Figure 1. This multiplication gain register is located between the end of the standard readout register and the output node of the device. A “high” voltage of 40-50 V is applied to one of the electrodes in the gain register, creating a higher electric field between the electrodes. This high electric field accelerates electrons so that they can effectively slam into the silicon lattice, generating further electrons. Through this impact ionisation in the silicon, the number of electrons is increased, providing multiplication gain in the charge domain before readout noise is added. The higher the voltage applied, the higher the electric field and therefore the higher the level of gain applied. Through increasing the gain whilst maintaining the same readout noise level, it is possible to reduce the effective read noise (compared to the input signal) to the sub-electron level.

In a standard CCD, the readout noise increases with increasing readout speed. The same applies in the case of the EM-CCD. However, with the addition of multiplication gain, one can simply increase the high voltage applied to counteract the increased noise and thus reduce it to the sub-electron level compared to the input signal, Figure 2.

Due to the stochastic nature of multiplication gain, the process is not noise free; the variance on the input signal level increases with increasing gain. The factor of the increase in the variance is defined by the Excess Noise Factor (ENF) [12]. The ENF is one at unity gain, increasing to a value of two at high gains (above 10-20 times gain), such that at high gains the noise increases by a factor of $\sqrt{2}$, Figure 3.

3. PROOF-OF-PRINCIPLE

To demonstrate the improved performance that is achievable through the use of photon-counting and centroiding, we developed a proof-of-principle camera [5-9]. The system was based around an e2v CCD97 [13], an EM-CCD with 512 × 512 pixels, each 16 µm square, giving an image area of approximately 8 mm × 8 mm. The camera was operated at approximately 2 fps. Whilst these specifications do not lend themselves to many applications, the system was designed to demonstrate the spatial and spectral resolution possibilities achievable with centroiding and is therefore independent of imaging area and, to a first approximation if using an EM-CCD, independent of the readout speed.

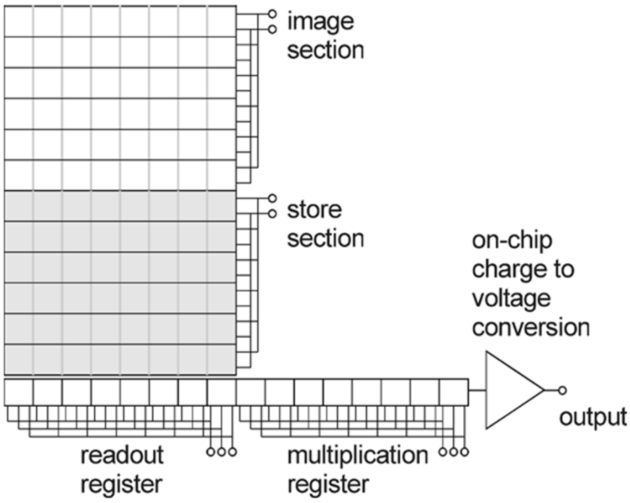

Figure 1. The Electron-Multiplying (EM) CCD, showing the addition of the multiplication register between the readout register and the output node [12].

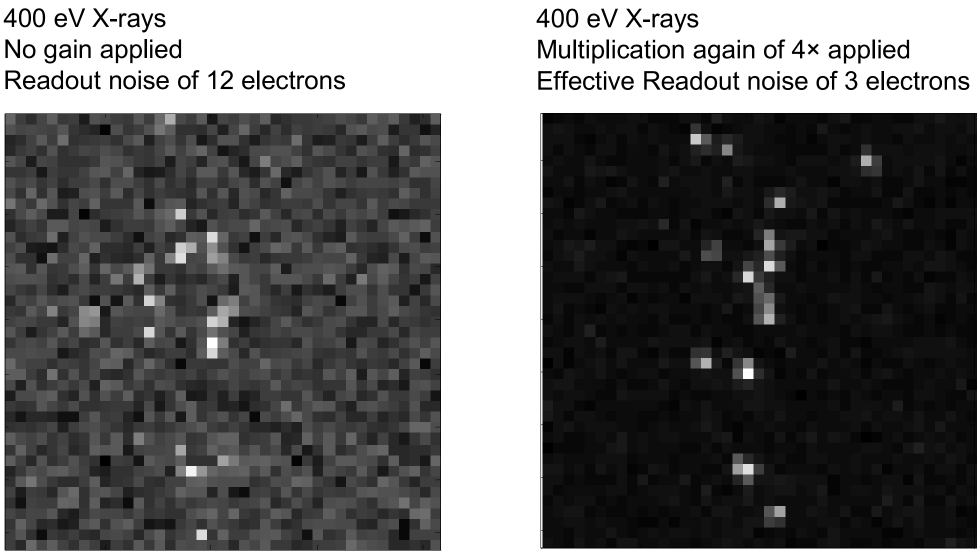

Figure 2. Example images taken with a CCD97 using 400 eV X-rays demonstrating the benefits of even small levels of multiplication gain, in this case where the initial noise is not overly high.

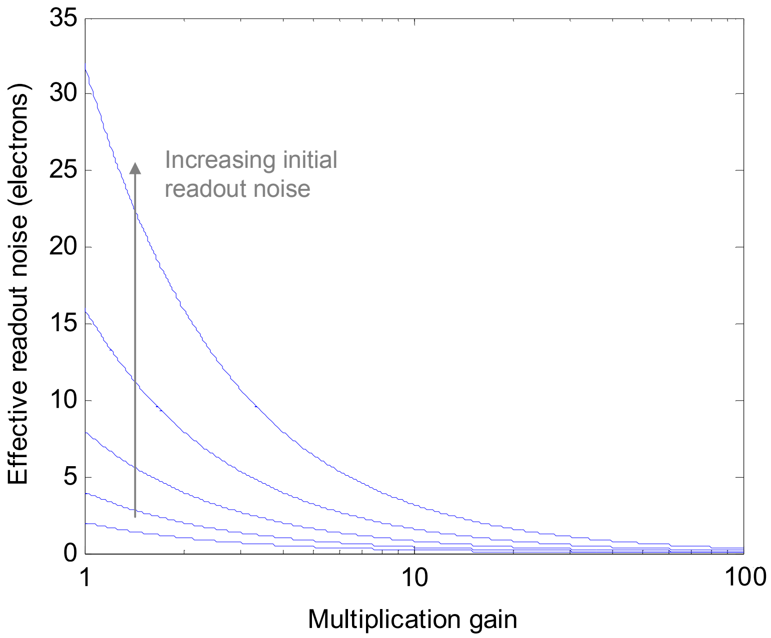

Figure 3. The effective readout noise (i.e. the readout noise as compared to a fixed signal level) decreases as the multiplication gain is increased. As the readout speed of a CCD or EM-CCD is increased, the readout noise also increases. However, with an EM-CCD, this increase in noise can be counteracted by modest levels of multiplication gain to maintain sub-electron effective readout noise.

As the quantum efficiency of the EM-CCD is very low for high energy X-rays (e.g. 20 keV to 200 keV), a scintillator was coupled to the detector through a 1:1 fibre-optic plate. The scintillator used was columnar-grown Caesium Iodide (Thallium doped) of 70 µm thickness. The scintillator is grown to have approximately 5 µm to 10 µm columns which act in a similar way to a fibre-optic in channelling light produced towards the surfaces of the scintillator. Although this process is not without its flaws, it provides a higher signal at the detector than would be achieved with a solid block of the same scintillator. Some of the light produced in the scintillator from the interaction of an incident X-ray photon spreads out radially (to a first approximation) from the point of interaction, despite the columnar structure. Each single X-ray photon therefore produces a spread of light at the detector, spreading over approximately 5 × 5 pixels to 9 × 9 pixels in area, with the peak intensity and the spread dependent on the X-ray energy and the interaction depth in the scintillator (a higher peak intensity and smaller spread would be seen with an X-ray interacting closer to the detector) [8,14,15].

A standard camera takes an "integrated" image; the shutter is opened and light incident on the detector builds up until it is read out. Using this method, the spread of light in the scintillator is included in the image itself, degrading the spatial resolution and removing the possibility of regaining any spectral information. If, however, one takes many images at high speed (or reduces the flux) such that each X-ray photon can be distinguished in the detector independently from all others, it is possible to centroid these events, removing the spread of light in the scintillator from the images and therefore improving the spatial resolution. Using this method of photon-counting, it is also possible to regain a limited degree of spectral information relating to the incident X-ray photons.

Using ideas developed from scale-space theories in computer vision [16-19], a novel analysis technique was developed [7,8]. By analysing each event with scale-space techniques, it is possible to take into account such parameters as the depth of interaction effects and provide useful spectral information about the incident signal. This analysis then allows fluorescence X-rays from surrounding materials to be removed (within limits) as well as those produced inside the scintillator that then interact and are detected independently (so called self-fluorescence).

Through the use of photon-counting and various centroiding techniques, this proof-of-principle system was able to demonstrate a dramatic improvement in the spatial resolution, Figure 4. Using integrating techniques a spatial resolution

(FWHM) was measured at 80 µm. With exactly the same experimental set-up, the centroiding techniques provided a spatial resolution 31 µm. However, with further spectral processing to remove both external and internal fluorescence, this resolution was improved by an extra 20% down to 25 µm (FWHM) [6], Table 1.

This dramatic improvement in spatial resolution through the use of centroiding and spectral analysis techniques demonstrated the potential of such a detector development. The small-area and low-speed were therefore the main limits to the use of the camera in terms of possible applications. With the promise shown, a large-area, high-speed variant was considered.

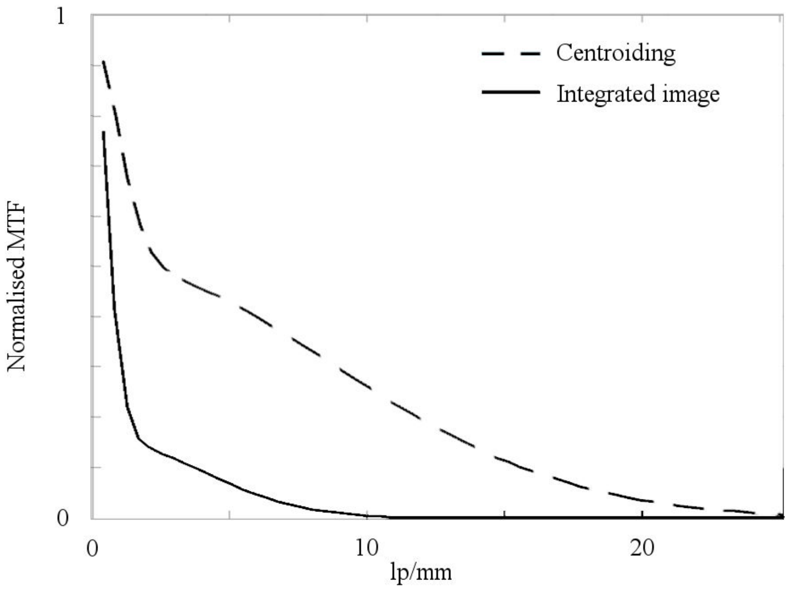

Figure 4. The MTF measured from a tungsten edge for the small-area proof-of principle camera system described in the text, demonstrating the dramatic improvement in the resolution when using photon-counting and centroiding techniques [5].

Table 1. The spatial resolution (FWHM) measured with the small-area proof-of principle camera system described in the text in three operating modes. Basic centroiding techniques show a strong improvement in the spatial resolution. With further analysis in the spectral-domain, both external and internal fluorescence can be removed to further improve the resolution [6].

Imaging method	**FWHM of LSF**
Integration (standard)	80 µm
Photon-counting with centroiding (basic)	31 µm
Photon-counting with centroiding (with removal of external and internal fluorescence through spectral analysis)	25 µm

4. SCINTILLATOR-COUPLED FIBRE-OPTIC TAPER SYSTEM

In order to increase the area of the detector two changes have been made from the initial system. The first is to use a larger area EM-CCD, the e2v CCD201. The second is to use a fibre-optic taper that can be coupled between the scintillator and EM-CCD. Furthermore, there are future plans to tile the single camera modules into a larger area array; however, here we outline the main component changes from the proof-of-principle study used to create the first single module and the impact that these changes will have system.

The scintillator remains CsI(Tl), which has again been chosen to its highly desirable properties such as high light-output and relatively good stopping power. Slightly thicker scintillating layers are being considered in this study, with approximate thickness of 135 µm. This increased thickness provides higher detection efficiency which will allow the system to be used at higher energies although the spreading of the light will be increased at low energies. How this affects the resolution achieved will be investigated in further studies. Two versions of the scintillator will be used with different coatings: one designed for high-resolution imaging with an effectively lower light output and one for high detection efficiency but with a lower spatial resolution achievable.

The e2v CCD201 [20] is similar in design to the CCD97 used in the proof-of-principle system described in Section 3. The CCD201 is a frame-transfer device and has 1024 × 1024 pixels in the image area, each 13 µm square, giving an image area of 13.3 mm × 13.3 mm, Figure 5. This in itself gives an imaging area increase of 75%, however, there are further implications. With an increase in the number of pixels by a factor of 4, the subsequent readout times also increase by the same factor for the same pixel readout rate. This increase in the readout time, coupled with the previously mentioned desire to operate at a higher frame-rate leads, to the requirement for high-speed drive electronics.

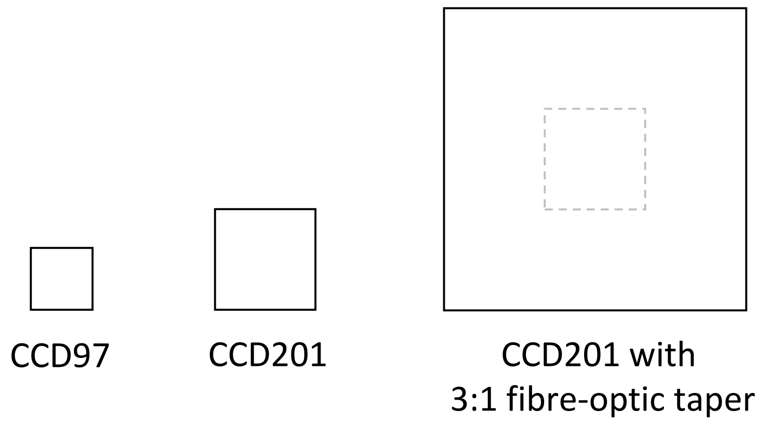

Figure 5. Demonstration of the image area sizes of the small-area proof-of-principle camera (left), the base EM-CCD for the new system (centre) and the system under development using a 3:1 fibre-optic taper (right).

To further increase the image area of the camera, the scintillator is coupled to the detector through a 3:1 fibre-optic taper instead of through a 1:1 fibre-optic plate as in the proof-of-principle system, Figure 5, providing an increase in imaging area over the previous proof-of-principle study of approximately 25 times. In the future coupling several camera modules together, in a 3 × 3 array for example, brings the ability to further increase the image area by over 200. During the development phase, a fibre-optic plate is coupled to the CCD201 and a second to the scintillator. Between the two fibre-optic plates is inserted the fibre-optic taper, allowing the components of the system such as the scintillator and taper size to be interchanged during testing. In the final system, the CCD201 and scintillator will be coupled directly to the small and large ends of the taper respectively.

The final main change to the components of the base camera is in the drive electronics system used. Whilst the proof-of-principle study was able to be performed at 2 fps (equivalent to 2 seconds per frame if used with the larger CCD201), we now aim to demonstrate the applicability of the camera module to more applications. To this end, we are using a very high-speed, low noise EM-CCD drive system from XCAM Ltd. [21], the Scientific CCD Camera System CCD6200 [11]. The system operates at 25 MHz, a large improvement on the 500 kHz readout rate previously used, and gives controllable and stable gain allowing sub-electron effective readout noise with the CCD201 at up to 30 fps (full-frame) or 120 fps with (quarter frame).

5. SYSTEM DEVELOPMENT

The cooling system used with the camera is TEC-based, with cooling capabilities reaching below -40°C when used alongside water cooling at approximately 10°C. The cooling is required to reduce the dark current and the associated noise on the dark current signal level. With the camera system able to operate at 30 fps full frame, the minimum frame time is therefore 1/30th of a second and this level of cooling is sufficient for the application.

The camera module currently under development will operate as a stand alone system, with the EM-CCD, taper and scintillator combination held under vacuum in a small chamber. The chamber is fronted with a custom window with 50% transmission above 2 keV up to approximately 100% transmission at 5 keV and above. Although the camera can be coupled to an external vacuum chamber with no window, it is envisaged that the majority of the testing for most applications will be performed in the standalone chamber.

The system is operating at 25 MHz pixel readout rate with a sub-electron system noise when gain is applied. Gain is stable to within approximately 3% over time (i.e. during use and after days not in operation) and is controllable by the user through the camera control system supplied with the system.

The proof-of-concept camera module is currently under development, with the first full taper-coupled module close to completion. Testing at Diamond on beamline B16 will take place in early December 2013 for several applications, including high-speed tomography and macromolecular crystallography.

During the system development, a short study was made into the possibility of using a scintillating fibre-optic plate, a perfectly structured scintillator which can have near-100% efficiency at 100 keV (with thickness over 1 cm) and formed from a scintillating glass. Although the resolution improvements predicted would be highly desirable, the significantly lower light output in comparison to that of CsI(Tl) led to the choice of structured CsI(Tl) for the current system development.

6. OPERATING MODES

Alongside the system development, we will be investigating various modes of operation relevant to specific applications. The flexibility of variable gain may provide many benefits to synchrotron users, allowing application-specific operation modes to further improve the detector performance.

For example, in macromolecular crystallography, the strong low resolution reflections can be measured at low gain such that the full well capacity of the device remains high in terms of incident signal. The increased frame-rate of the camera will effectively allow signal handling 30 times greater at 30fps than a standard CCD-based system can offer with current readout rates of the order of 1 fps at full-frame (no binning) [3,4]. For weak, high resolution reflections, the gain can be increased, reducing the effective readout noise to the sub-electron level and allowing photon-counting and centroiding techniques to be used to improve the spatial resolution. The reduced full well capacity (in terms of the incident signal level) with high gains will not be a problem for the weak reflections with low signals. The system will be tested across each range of application-specific modes to ensure optimisation across a range of conditions. The projected performance of the system will also offer other benefits, including higher throughput in the beamline facilities through shutter-less performance using frame-transfer devices and high-speed readout.

7. DIRECT DETECTION

Although the camera system under development is primarily directed towards the detection of hard X-rays (10 keV-200keV) using indirect detection through a scintillator, the system can also be operated for the direct detection of soft X-rays (0.1 keV-10 keV). Previous studies of the applicability of the EM-CCD for the International X-ray Observatory (IXO) [22] demonstrated the suitability of the detector for the Off-Plane X-ray Grating Spectrometer [23] in the energy range of 0.3 keV-1 keV.

In the field of synchrotron research it has been demonstrated that the EM-CCD can bring many benefits to spatially dispersive soft X-ray spectrometers [24-28]. The ability to operate the spectrometer in photon-counting mode due to the high-speed readout with sub-electron readout noise allows centroiding techniques to be used. The use of photon-counting and centroiding allows order of magnitude improvements to be made to the spatial resolution, impacting directly on the spectral resolution achievable with the spectrometer [24-28], both in ground-based applications and in space [29].

8. CONCLUSIONS

Current systems for the imaging of X-rays (10 keV-200 keV) in synchrotron research are generally based around scintillator-coupled CCDs or hybrid pixel detectors. Whilst both detector systems have their benefits, each has limitations. The aim of this system development is to produce a proof-of-concept camera module for synchrotron research, offering the best of both worlds: high-speed, high-resolution, low noise, shutter-less detection using an EM-CCD and fibre-optic taper, whilst improving on the detection efficiency, effective dynamic range and operating temperature of CCD-based systems.

We have previously demonstrated the benefits that the EM-CCD can bring to scintillator-coupled detector systems with a 1:1 fibre-optic plate. The use of centroiding with photon-counting images, alongside novel spectral analysis techniques, provided a resolution improvement from 80 µm to 25 µm. The current system development will provide a 25 MHz pixel readout rate system (approximately 30 fps full-frame or 120 fps quarter-image) whilst also providing a 25× increased image area. Performance details of the full system and in-situ test results will be presented in a future paper.

9. ACKNOWLEDGEMENTS

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